



**You have downloaded a document from
RE-BUS
repository of the University of Silesia in Katowice**

Title: Phase stability of $(\text{Fe}_{80}\text{Nb}_{6}\text{B}_{14})(0.9)\text{Tb}-0.1$ bulk nanocrystalline magnet

Author: Artur Chrobak, Grzegorz Ziółkowski, N. Randrianantoandro

Citation style: Chrobak Artur, Ziółkowski Grzegorz, Randrianantoandro N. (2014). Phase stability of $(\text{Fe}_{80}\text{Nb}_{6}\text{B}_{14})(0.9)\text{Tb}-0.1$ bulk nanocrystalline magnet. "Acta Physica Polonica A" (Vol. 126, nr 1 (2014), s. 176-177), doi 10.12693/APhysPolA.126.176



Uznanie autorstwa - Użycie niekomercyjne - Bez utworów zależnych Polska - Licencja ta zezwala na rozpowszechnianie, przedstawianie i wykonywanie utworu jedynie w celach niekomercyjnych oraz pod warunkiem zachowania go w oryginalnej postaci (nie tworzenia utworów zależnych).



UNIwersYTET ŚLĄSKI
W KATOWICACH



Biblioteka
Uniwersytetu Śląskiego



Ministerstwo Nauki
i Szkolnictwa Wyższego

Phase Stability of $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ Bulk Nanocrystalline Magnet

A. CHROBAK^{a,*}, G. ZIÓŁKOWSKI^a, N. RANDRIANANTOANDRO^b^aInstitute of Physics, University of Silesia, Uniwersytecka 4, 40-007 Katowice, Poland^bInstitut des Molécules et des Matériaux du Mans, UMR CNRS 6283, Université du Maine, 72085 Le Mans cedex 9, France

The paper refers to phase stability of the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ bulk nanocrystalline alloys prepared using the vacuum suction casting technique. The samples were in the form of rods with diameters $d = 2, 1.5, 1$ and 0.5 mm. Heating up to 900 K reveals structural changes that occur at temperatures above 680 K (DSC and $M(T)$ measurements). The phase analysis, using Mössbauer spectra, indicates the decrease of $\text{Tb}_2\text{Fe}_{14}\text{B}$ and increase of Fe content in the samples after the heat treatment. The most stable is the alloy with $d = 1$ mm, where the formation of α -Fe phase was not observed. The decrease of d causes significant hardening i.e. coercive field increases from 0.57 T to 2.66 T for $d = 2$ mm and $d = 0.5$ mm, respectively.

DOI: [10.12693/APhysPolA.126.176](https://doi.org/10.12693/APhysPolA.126.176)

PACS: 81.07.Bc, 75.50.Tt, 75.60.d, 76.80.+y

1. Introduction

Progress in modern technologies requires new materials with specific properties for different kind of applications [1, 2]. In the field of magnetism very interesting are Fe-Nb-B type of nanocrystalline alloys [3, 4]. Recently we have reported that the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{1-x}\text{Tb}_x$ bulk nanocrystalline series of alloys can be considered as highly coercive materials [5, 6]. It was shown (^{57}Fe Mossbauer spectrometry and XRD) that the examined samples contain magnetically hard $\text{Tb}_2\text{Fe}_{14}\text{B}$ and other TbFe_2 , α -Fe soft phases, high Tb content the high contribution of TbFe_2 . The samples with $x = 0.1$ (69% of $\text{Tb}_2\text{Fe}_{14}\text{B}$) and $x = 0.12$ (76% of $\text{Tb}_2\text{Fe}_{14}\text{B}$) are interesting. For these samples the magnetic hardening is significant, i.e. at $T = 300$ K, $H_c = 1.46$ T and 1.16 T, respectively. It is a characteristic feature that nanocrystalline alloys are not thermodynamically stable and, during annealing, some phase transitions (or separations) are expected to be present [7]. Therefore, in this case phase stability studies are important from application as well as scientific point of view. In this work we present structural and magnetic properties of $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ bulk nanocrystalline alloy in the as-cast state and after heating up to 900 K.

2. Experimental procedure

Samples of the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ alloy were prepared by means of the vacuum suction casting technique in the form of rods with diameters $d = 2, 1.5, 1$ and 0.5 mm (described in [8]). In the technology the variation of d causes a change of cooling. The alloys were examined before and after heating up to 900 K. Phase

changes were studied using NETZSCH scanning differential calorimetry (heating rate 20 K/min), ^{57}Fe Mössbauer spectrometry (in transmission geometry with constant acceleration spectrometer, using a ^{57}Co source diffused in a rhodium matrix) and magnetic measurements (SQUID magnetometer Quantum Design XL-7 and Faraday type magnetic balance).

3. Results and discussion

Figure 1 shows an example of the DSC and thermomagnetic $M(T)$ measurements for the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ alloy with $d = 1.5$ mm. The magnetization curve clearly indicates the Curie temperature T_c (related to the $\text{Tb}_2\text{Fe}_{14}\text{B}$ phase) and structural changes (the increase of M above 680 K). The observed magnetization increase is attributed to formation of a magnetic phase with higher T_c .

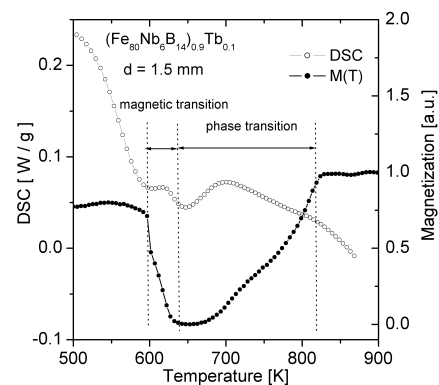


Fig. 1. DSC and $M(T)$ curves for the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ alloy with $d = 1.5$ mm.

The DSC curve reveals two exothermic peaks ascribed to the magnetic transition and phase transition (or changes). Similar results were obtained for the other

*corresponding author; e-mail: artur.chrobak@us.edu.pl

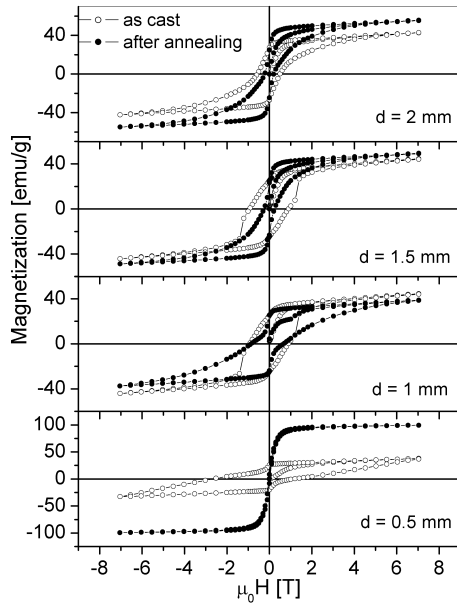


Fig. 2. Magnetic hysteresis loops measured at $T = 300$ K for the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ alloy with different d (before and after annealing).

examined samples with different d . Phase detection was performed based on ^{57}Fe Mössbauer spectrometry (not shown here) recorded at room temperature. For all studied cases the spectra were deconvoluted into a set of elementary Zeeman sextets (attributed to different phases) by a least-square fit procedure. The results are presented in Table.

Let's note that, except the sample with $d = 1$ mm, a decrease of $\text{Tb}_2\text{Fe}_{14}\text{B}$ and increase of $\alpha\text{-Fe}$ content after annealing were observed.

Figure 2 shows magnetic hysteresis loops measured at $T = 300$ K for all studied alloys before and after annealing.

TABLE

Phase content analysis (percentage), determined from Mössbauer spectra for the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ alloy. (A – as cast, B – after annealing).

d (mm)	$\text{Tb}_2\text{Fe}_{14}\text{B}$		TbFe		$\alpha\text{-Fe}$		Paramagn.	
	A	B	A	B	A	B	A	B
0.5	75	17	9	14	-	53	16	16
1	65	69.5	15	15.5	-	-	14	15
1.5	62	56	15	11	6	17	17	16
2	64	56	18	11	-	17	18	16

As it is shown, the annealing causes a deterioration of hard magnetic properties (decrease of the coercive field). Apart from the main subject, for the as-cast alloys one can observe a significant magnetic hardening as a function of d (or cooling rate during sample fabrication) i.e. H_c increases from 0.57 T to 2.66 T for $d = 2$ mm and $d = 0.5$ mm, respectively. This effect was studied in

details in a separate work [9]. Generally, the change of magnetic properties is related to the phases variation. The less stable is the alloy with $d = 0.5$ mm, where the significant decrease of $\text{Tb}_2\text{Fe}_{14}\text{B}$ and separation of $\alpha\text{-Fe}$ was observed. In contrast to this, the most stable is the alloy with $d = 1$ mm, where in the as-cast state, as well as after heating, the formation of $\alpha\text{-Fe}$ was not observed. This fact has a practical meaning i.e. the annealed alloy is thermodynamically stable and possesses a good potential as permanent magnet for high temperature applications (PM motors, actuators, sensors etc.).

4. Conclusions

Referring to the $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ bulk alloys the main conclusions can be summarized as follows.

Heating up to 900 K reveals some structural changes that occur at temperatures above 680 K (DSC and $M(T)$ measurements). The performed phase analysis, using ^{57}Fe Mössbauer spectrometry, indicates the decrease of $\text{Tb}_2\text{Fe}_{14}\text{B}$ and increase of $\alpha\text{-Fe}$ content for the heat treated samples. The most stable is the alloy with $d = 1$ mm, where the formation of $\alpha\text{-Fe}$ phase was not observed.

Regarding magnetic properties, the decrease of d causes significant hardening, i.e. the coercive field increases from 0.57 T to 2.66 T for $d = 2$ mm and $d = 0.5$ mm, respectively. The heating leads to the observed deterioration of hard magnetic properties, especially for the alloy with $d = 0.5$ mm. However for $d = 1$ mm, the alloy after annealing is quite stable and can be use in some high temperature applications.

Acknowledgments

Grzegorz Ziółkowski acknowledges a scholarship from the TWIG project co-financed by the European Social Fund.

References

- [1] M.E. McHenry, M.A. Willard, D.E. Laughli, , *Prog. in Mat. Sci.* **44**, 291 (1999).
- [2] K.H.J. Buschow, F.R. de Boer, *Physics of magnetism and magnetic materials*, Kluwer Academic Publishers, 2004.
- [3] M. Sagawa, S. Fujimura, H.i Yamamoto, Y. Mat-suura, *IEEE Trans. Magn.* **20**, 1584 (1984).
- [4] N. Randrianantoandro, A.D. Crisan, O. Crisan, J. Marcin, J. Kovac, J. Hanko, J.M. Grenèche, P. Svec, A. Chrobak, I. Skorvanek, *J. Appl. Phys* **108**, 093910 (2010).
- [5] G.Ziółkowski, N.Randrianantoandro, A.Chrobak, J.Klimontko, M.Kądziołka-Gaweł, G.Haneczok, *Acta Phys. Pol. A* **121**, 1266 (2012).
- [6] A.ChrobakG. Ziółkowski, N. Randrianantoandro, J. Klimontko, G. Haneczok, *J. Alloys Compd.* **537**, 154 (2012).
- [7] T. Kulik, *J. Non-Cryst. Solids*, **287**, 145 (2001).
- [8] A.Chrobak, M.Karolus, G.Haneczok, *Solid State Phenomena* **163**, 233 (2010).
- [9] A. Chrobak, G. Ziółkowski, N. Randrianantoandro, *J. Alloys Compd.* **583**, 48 (2014).